

NOTE: After the sample and colorimetric reagent are mixed, the color formed is stable for approximately 2 hours. Also, a 3 °C (5.4 °F) temperature difference between the sample and standard solutions produces an error of approximately 0.005 mg F⁻/liter. To avoid this error, the absorbencies of the sample and standard solutions must be measured at the same temperature.

11.4.1.3 Set the spectrophotometer to zero absorbance at 570 nm with the zero reference solution (Section 7.3.12), and check the spectrophotometer calibration with the standard solution (Section 7.3.10). Determine the absorbance of the samples, and determine the concentration from the calibration curve. If the concentration does not fall within the range of the calibration curve, repeat the procedure using a different size aliquot.

11.4.2 Container No. 3 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after final calculation. Other forms of the equations may be used, provided that they yield equivalent results.

12.1 Nomenclature.

A_d = Aliquot of distillate taken for color development, ml.
 A_t = Aliquot of total sample added to still, ml.
 B_{ws} = Water vapor in the gas stream, portion by volume.
 C_s = Concentration of F⁻ in stack gas, mg/dscm (gr/dscf).
 F_c = F⁻ concentration from the calibration curve, µg.
 F_t = Total F⁻ in sample, mg.
 T_m = Absolute average dry gas meter (DGM) temperature (see Figure 5-3 of Method 5), °K (°R).
 T_s = Absolute average stack gas temperature (see Figure 5-3 of Method 5), °K (°R).
 V_d = Volume of distillate as diluted, ml.
 $V_{m(std)}$ = Volume of gas sample as measured by DGM at standard conditions, dscm (dscf).
 V_t = Total volume of F⁻ sample, after final dilution, ml.
 $V_{w(std)}$ = Volume of water vapor in the gas sample at standard conditions, scm (scf)

12.2 Average DGM Temperature and Average Orifice Pressure Drop (see Figure 5-3 of Method 5).

12.3 Dry Gas Volume. Calculate $V_{m(std)}$, and adjust for leakage, if necessary, using Equation 5-1 of Method 5.

12.4 Volume of Water Vapor and Moisture Content. Calculate $V_{w(std)}$ and B_{ws} from the data obtained in this method. Use Equations 5-2 and 5-3 of Method 5.

12.5 Total Fluoride in Sample. Calculate the amount of F⁻ in the sample using the following equation:

$$F_t = \frac{K V_t V_d F_c}{A_t A_d} \quad \text{Eq. 13A-1}$$

Where:

$K = 10^{-3}$ mg/µg (metric units)
 $= 1.54 \times 10^{-5}$ gr/µg (English units)

12.6 Fluoride Concentration in Stack Gas. Determine the F⁻ concentration in the stack gas using the following equation:

$$C_f = \frac{F_t}{V_{m(std)}} \quad \text{Eq. 13A-2}$$

12.7 Isokinetic Variation. Same as Method 5, Section 12.11.

13.0 Method Performance

The following estimates are based on a collaborative test done at a primary aluminum smelter. In the test, six laboratories each sampled the stack simultaneously using two sampling trains for a total of 12 samples per sampling run. Fluoride concentrations encountered during the test ranged from 0.1 to 1.4 mg F⁻/m³.

13.1 Precision. The intra- and inter-laboratory standard deviations, which include sampling and analysis errors, were 0.044 mg F⁻/m³ with 60 degrees of freedom and 0.064 mg F⁻/m³ with five degrees of freedom, respectively.

13.2 Bias. The collaborative test did not find any bias in the analytical method.

13.3 Range. The range of this method is 0 to 1.4 µg F⁻/ml.

14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Alternative Procedures

16.1 Compliance with ASTM D 3270-73T, 80, 91, or 95 (incorporated by reference—see §60.17) “Analysis of Fluoride Content of the Atmosphere and Plant Tissues (Semiautomated Method) is an acceptable alternative for the requirements specified in Sections 11.2, 11.3, and 11.4.1 when applied to suitable aliquots of Containers 1 and 2 samples.

17.0 References

1. Bellack, Ervin. Simplified Fluoride Distillation Method. J. of the American Water Works Association. 50:5306. 1958.
2. Mitchell, W.J., J.C. Suggs, and F.J. Bergman. Collaborative Study of EPA Method 13A and Method 13B. Publication No. EPA-300/4-77-050. U.S. Environmental Protection Agency, Research Triangle Park, NC. December 1977.